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TestAmerica, Inc.

Dayton Division

US EPA RECORDS CENTER REGION 5
471492

Standard Operating Procedure

Analyte or Suite: 1,2-Dibromoethane/1,2-Dibromo-3-chloropropane

Methodology: Microextraction/Gas Chromatography

Reference: USEPA Method 504.1 Revision 1.1

Revision # DT06-13.1 Date Revised: October 20, 1999

Approvals:

B. Wall
Divisional Manager

James A. Davis
Quality Assurance
Coordinator

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1. INTRODUCTION AND SCOPE

1.1. General

Preservative: For chlorinated systems add Sodium thiosulfate otherwise store at 4 degrees C until analysis.

Container: 40mL vials with teflon-lined septa

Minimum sample volume: 40 ml vial

Holding Time: 14 days ; Extracts: 24 hours

Reporting Limit: <0.02 ug/L

2. SUMMARY OF METHOD

This method provides extraction, preparation, and chromatographic conditions for the detection of EDB and DBCP at sub ug/L levels.

2.1. The sample is extracted with hexane using microextraction in the sample container. The hexane extract is removed, bottled and is ready for analysis by capillary GC/ECD.

3. SAFETY

Each employee is directly responsible for complete awareness of all health hazards associated with every chemical that he/she uses. The employee must be aware of these hazards, and all associated protective wear and spill clean-up procedures PRIOR TO THE USE of any chemical. In all cases, both the applicable MSDS and supervisor or Safety Officer should be consulted. The employee should comply with all safety policies as presented in the TestAmerica Safety Manual. The bottle labels also provide important information that must be noted. Personnel performing this procedure may be working with flammables, poisons, toxins, carcinogens, teratogens, mutagens, and biohazards. In particular, approved gloves, safety glasses, and labcoats must be worn, and solvents will be handled in ventilated hoods, in addition to other measures prescribed by the Division. It should be noted that samples must be handled with as much (or more) care as any of the materials used in this method due to the unknown mature of their composition. Also, the equipment utilized by this method contain areas of both high temperature and potentially lethal voltage. Care must be taken whenever performing maintenance on these systems.

4. REAGENTS AND MATERIALS

The following equipment and materials (or their equivalent) are recommended for this method. Equipment and materials are considered equivalent if with their use, the analytical and QA/QC requirements in this SOP can be met.

4.1. Apparatus

4.1.1. Microsyringes - gas tight 10uL, 25uL, 100uL and 250uL.

Whenever possible, choose the syringe size that maximizes the volume usage of the syringe for the most effective results.

4.1.2. Volumetric flasks - 50mL and 100mL; Class A

4.1.3. Volumetric pipets, 2mL; Class A

4.1.4. Disposable pipets - Pasteur.

4.1.5. 40mL volatile sampling bottles with teflon lined septa.

4.2. Gas Chromatography

4.2.1. Gas Chromatograph, capillary GC capable of autosampling with all accessories, including columns, gases, vials, and syringes. The instrument and associated data acquisition system must be capable of meeting the quality control requirements set forth in this procedure.

4.2.2. Columns:

Column A: (Primary Column)
SPB-608 30 X 0.25 mm ID or equivalent

Column B: (Confrimation Column)
PTE-5 30 X 0.25 mm ID or equivalent

4.3. Reagents

4.3.1. Reagent grade chemicals should be used in all tests.

4.3.2. Reagent water. Must not contain interferences at or above the RL.

4.3.3. Sodium choride, reagent grade. Bake at 400 degrees C for 30 minutes and store in a bottle with teflon lined cap.

4.3.4. Sodium Thiosulfate, reagent grade.

4.3.5. Hexane, pesticide grade. Caution: Hexane is extremely flammable. Keep it away from heat, sparks, and flames. Causes irritation. Toxic by ingestion and inhalation. Avoid inhalation by working with hexane in a fume hood. Gloves and safety glasses must be worn to avoid contact with the skin and eyes.

4.4. Standards

Standards and spiking solutions are prepared from either assayed reference materials or certified solutions.

4.4.1. Calibration Standards are prepared prior to injection. Prepare standards at the following levels:

Cal 1	0.02ug/L
Cal 2	0.04ug/L
Cal 3	0.10ug/L
Cal 4	0.20ug/L
Cal 5	0.40ug/L

5. INTERFERENCES

5.1. Careful screening of potential lots of extraction solvent will generally eliminate solvent based interference. Extraction solvent is analyzed frequently in addition to the regular monitoring of reagent water blanks. Dibromochloromethane may also mask EDB in sample chromatograms if present at high levels.

5.2. All positive detections should be confirmed by GCMS especially EDB as dibromochloromethane (DBCM) may interfere.

6. ANALYTICAL PROCEDURE

6.1 SAMPLE COLLECTION, PRESERVATION AND STORAGE

Chlorinated volatile samples are preserved with sodium thiosulfate and stored at 4 degrees C until anlaysis. Samples must be extracted and analyzed within 14 days of collection.

6.1.2 The samples must be chilled to 4 C on the day of collection and maintained at that temperature until analysis. Samples that will not be received at the laboratory on the day of collection must be packaged for shipment with sufficient ice to ensure they will be at 4 C on arrival at the laboratory.

6.1.3 Sample extracts may be stored in tightly capped vials at 4 degrees C or less for up to 24 hours.

6.2 Recommended GC Operating Conditions

Gas Chromatography Conditions

Columns: SPB-608 and PTE-5

Injection volume:	3.	uL
Initial oven temperature:	65.	C
Initial oven hold time:	3.	minutes
Oven temp rate:	6.	C/minute
Oven temp final value:	180.	minutes
Oven temp final hold time	2.	minutes

6.3 Initial Calibration

6.3.1. Prepare calibration standards as indicated in Standards Section and load each of the calibration levels into a specified position on the autosampler. A reagent blank is analyzed after the calibration to ensure the system is reasonably free of contamination. An Initial Calibration Verification standard (ICV) is analyzed after an acceptable curve is generated to ensure the curve's accuracy.

6.3.2. For each analyte, calculate the mean RF for the analyses of the calibration solutions. The response factor is calculated by dividing the concentration of the analyte by the area of the peak.

$$\text{Response Factor} = \frac{\text{Conc. of analyte}}{\text{Area of peak}}$$

Calculate the standard deviation (SD) and the relative standard deviation (RSD) from each mean: RSD = 100 (SD/M). If the RSD of any analyte exceeds 20%, either analyze additional aliquots of appropriate calibration solutions to obtain an acceptable RSD of RFs over the entire concentration range, or take action to improve the GC performance.

6.3.3. After a successful initial calibration, measures must be taken to ensure that the system is contaminate free. Analyze a reagent DI water blank to evaluate the system. The blank must below the reporting limit for all compounds.

6.3.4. The data that is generated from an acceptable calibration curve is quantitated off of the average response factor from that calibration curve.

6.3.5. The analysis of an independent reference standard, or ICVS is required immediately following each curve. The ICVS is prepared at the same level as Cal#4 and quantitated off of the average response factor generated from the calibration curve. If the quantitation results are not within +/- 25 percent of the true value notify your supervisor. The +/- 25 percent is a warning limit. Document all ICVS results.

6.4. Sample Preparation/Microextraction

6.4.1. Remove approximately 5 mL from the sample bottle and discard the removed portion. Weigh the capped sample and record the weight to one decimal place.

6.4.2. Add 6 grams of sodium chloride to the sample remaining in the bottle, cap and shake for approximately 20 seconds (or as needed) to dissolve the sodium chloride.

6.4.3. Next add 2.0mL of hexane using a volumetric pipet or automatic dispensing pipet seal and shake vigorously for approximately 1 minute.

6.4.4. Allow the sample to rest inverted for at least five minutes. Note: If an extracted sample is to sit for a length of time, it should be stored inverted to prevent the loss of hexane due to evaporation.

6.4.5. Pipet off 1 mL of the hexane extract and bottle in a 2 mL GC autosampler vial. The extract is now ready for analysis by GC/ECD.

6.4.6. The sample bottle is emptied and reweighed to determine sample volume assuming a water density of 1 gram/cc.

6.5. Sample Analysis

6.5.1. GC Sample Reports

6.5.1.1. The GC operator shall set up the sequence by entering

into the run log a listing of sample numbers in order of analysis.

6.5.2. Qualitative Analysis.

6.5.2.1. An analyte is identified by comparison of the sample retention time to the reference retention time. If individual retention times vary by more than +/- 0.08 minutes from the average retention time of the calibration, the system is recognized as out of control. The retention time variation must be corrected before continuation of sample analysis. NOTE: An alternate procedure for determining retention time windows would be to use 3 times the standard deviation determined from the average retention of the initial calibration. To be considered a legitimate hit the analyte must show up in it's expected retention time range on both columns.

6.5.2.2. Calculate the concentration of the samples by comparing the peak areas of the samples to the standard peak areas.

Concentration (ug/L) = Average RF of compd. X Area of peak

7. Quality Control

The laboratory must maintain records to document the quality of the data generated and these records will be regularly audited. The record must be complete and well organized. On-going data quality checks are compared with established performance criteria to determine if the results of the analyses meet the performance criteria for the method or alternatively, those generated in-house,provided they meet the minimum performance specification of this method.

The experience of the analyst performing GC analyses is invaluable to the success of the method. Each day that analysis is performed, the daily calibration standard must be evaluated to determine if the chromatographic system is operating properly. Questions that should be asked are: Do the peaks look normal? Is the response obtained comparable to the response from previous calibrations? Careful examination of the standard chromatogram is critical to identify any problems with the GC system. If any major changes are done to the system, such as a column change recalibration must take place.

7.1. Quality control requirements.

Quality control (QC) requirements are the initial demonstration of laboratory capability followed by regular analyses of laboratory reagent blanks and continuing calibration verification standards. Each laboratory must maintain records to document the quality of the data generated.

7.1.1. Method Blank.

Initial demonstration of low system background. Before any samples are analyzed, it must be demonstrated that a laboratory reagent (DI water blank) is reasonably free of contamination that would prevent the determination of any analyte of concern.

Background contamination must be reduced to an acceptable level before proceeding with the next section. In general, background from method analytes should be below the method reporting limits.

7.1.2. Initial Calibration Curve.

A calibration curve is run when the CCV fails to meet performance criteria or when there is a change in analyte, instrument, or standard materials. A recommended 5 point curve should be performed with a minimum of 3 points mandatory. An ICV will be run along with the curve to verify the curve.

7.1.3. Initial Calibration Verification (ICV/LFB)

An ICV is analyzed following a new calibration curve to validate the curve. The ICV is from a different source than the calibration standards. This standard also serves as the Laboratory Fortified Blank (LFB) that has been spiked with EDB and DBCP at 0.20 ug/L. The percent deviation of the LFB must be no greater than +/- 30%

7.1.4. Continuing Calibration Verification (CCV).

The initial calibration must be verified every 12 hours by the analysis of one CCV at any of the calibration levels stated in 4.4.1.

7.1.5. Laboratory Reagent Blank (LRB).

The laboratory reagent blank is deionized water that is subjected to the same conditions that a prepared sample undergoes. One LRB is extracted and analyzed per 20 samples extracted. Acceptance criteria requires that the reagent blank be less than the reporting limit.

7.1.6. MS/MSD.

The matrix spike and matrix spike duplicate pair are two separate aliquots of sample which are spiked with known concentrations of analyte and subjected to the same conditions that a sample undergoes. Analyze a minimum of one MS/MSD pair per every 20 samples. Advisory interim acceptance criteria requires the MS/MSD percent recovery to be within 65-135%. Precision between the MS and MSD needs to meet advisory limits of <25%

7.1.7. Reporting Limit Verification Standard (RLVS)

The RLVS is extracted from an aliquot of deionized water which has been spiked with EDB and DBCP at 0.02ug/L. It must be extracted and analyzed weekly to determine whether the instrument and method are capable of achieving the stated reporting limits.

8. REFERENCES

1. U.S. Environmental Protection Agency, "1,2-Dibromoethane and 1,2-Dibromo-3-Chloropropane in Water by Microextraction and Gas Chromatography" Enviromental Monitoring Systems Laboratory, U.S.E.P.A. Concinnati, Ohio, (1995). Method 504.1 Revision 1.1

October 22, 1999

Mr. Jon Peterson
EPA Project Coordinator
U.S. Environmental Protection Agency
Region 5
77 West Jackson Blvd.
Chicago, IL 60604

RE: Submittal of Standard Operating Procedures for Test America, Inc. and Air Toxics Ltd. and Addendum to the Quality Assurance Program Plan for the Albion-Sheridan Township Landfill Superfund Site
ALB025.100.0012

Dear Mr. Peterson:

Hull & Associates, Inc. (HAI) has developed this letter on behalf of the Settling O&M Defendants (City of Albion & Decker Manufacturing) to submit to U.S. EPA Test America Inc.'s (TAI) and Air Toxics Ltd.'s (ATL) standard operating procedures (SOPs) to be utilized by during the O&M activities at the Albion-Sheridan Township Landfill Superfund Site. TAI is the analytical laboratory selected to perform the analytical analysis for the O&M program and the one-time TO-14 analysis will be performed by ATL.

This submittal also serves, upon approval, as an addendum to the Remedial Action - Quality Assurance Program Plan (QAPP, WWC, August 1997). The analytical SOPs supplied in the original QAPP were specific to Quanterra Laboratories, and as such, are not practical to TAI. TAI has supplied SOPs that are equivalent in methodology and quality control/quality assurance procedures as those supplied in the original QAPP. Note that the gold colored pages in this submittal indicate specific changes to the QAPP that TAI is proposing for routine laboratory practices.

As per your discussion with Ms. Penny Davis of our office on October 14, 1999, the Settling O&M Defendants will proceed with the sampling event scheduled for the week of October 25, 1999 and will authorize TAI and ATL to perform analytical methodology in accordance with this submittal. If U.S. EPA requests any changes to the SOPs submitted by TAI, these changes will be incorporated prior to the completion of the second quarterly O&M sampling event (January 2000).

Please feel free to contact me at your convenience, should you have any questions or comments regarding the above information.

Sincerely,

William G. Petruzzi, P.G.
Senior Project Manager

WGP/pkd

ct: Chief, Environmental Enforcement Section, U.S. Department of Justice
Director, Superfund Division, U.S. EPA
Leroy Schmidt, City Engineer, City of Albion
Charles Denton, Varnum, Riddering, Schmidt & Howlett LLP
Philip M. Moilanen, Bullen, Moilanen, Klaasen & Swan
Mary Schafer, Michigan Department of Environmental Quality
Terry Baehr, Hull & Associates, Inc.